

Claims

1. A process for the production of acetic acid which process comprises the steps of
(1) carbonylating methanol and/or a reactive derivative thereof in a first carbonylation
reaction zone in a liquid reaction composition comprising iridium carbonylation
catalyst, ruthenium promoter, methyl iodide co-catalyst, methyl acetate, acetic acid and
5 water;
(2) withdrawing liquid reaction composition together with dissolved and/or entrained
carbon monoxide and other gases from said carbonylation reaction zone;
(3) optionally passing said withdrawn liquid reaction composition through one or more
further reaction zones to consume at least a portion of the dissolved and/or entrained
10 carbon monoxide;
(4) passing said composition from step (2) and optional step (3) into one or more flash
separation stages to form (i) a vapour fraction comprising condensable components and
low pressure off-gas, the condensable components comprising acetic acid product and
the low pressure off-gas comprising carbon monoxide and other gases dissolved and/or
15 entrained with the withdrawn liquid carbonylation reaction composition and (ii) a liquid
fraction comprising iridium carbonylation catalyst, ruthenium promoter and acetic acid
solvent;
(5) separating the condensable components from the low pressure off-gas; and
(6) recycling the liquid fraction from the flash separation stage to the first carbonylation
20 reaction zone,
wherein the concentration of carbon monoxide in the low pressure off-gas is maintained
according to the formula :

$$Y > mX + C$$

wherein Y is the molar concentration of carbon monoxide in the low pressure off-gas, X is the concentration in ppm by weight of ruthenium in the liquid reaction composition, m is about 0.012 and C is about -8.7.

2. A process according to claim 1 wherein the liquid reaction composition together
5 with dissolved and/or entrained carbon monoxide withdrawn from the first reaction zone is passed through a second reaction zone
3. A process according to claim 2 wherein substantially all the liquid reaction composition together with dissolved and/or entrained carbon monoxide withdrawn from the first reaction zone is passed to the second reaction zone.
- 10 4. A process according to any one of claims 1 to 3 wherein carbon monoxide in addition to that introduced to the second reaction zone as dissolved and/or entrained carbon monoxide is introduced into the second reaction zone
5. A process according to claim 4 wherein the additional carbon monoxide is co-joined with the first liquid reaction composition prior to introduction to the second
15 reaction zone and/or is fed separately to one or more locations within the second reaction zone.
6. A process according to claim 4 or 5 wherein the additional carbon monoxide contains impurities.
7. A process according to any one of claims 4 to 6 wherein the additional carbon
20 monoxide comprises high pressure off-gas from the first reaction zone.
8. A process according to any one of claims 4 to 6 wherein the additional carbon monoxide comprises a carbon monoxide-containing gas stream.
9. A process according to any one of claims 1 to 8 wherein greater than 10% of the dissolved and/or entrained carbon monoxide in the liquid reaction composition
25 withdrawn from the first reaction zone is consumed in the second reaction zone.
10. A process according to any one of claims 1 to 8 wherein greater than 25% of the dissolved and/or entrained carbon monoxide in the liquid reaction composition withdrawn from the first reaction zone is consumed in the second reaction zone.
11. A process according to any one of claims 1 to 8 wherein greater than 50% of the
30 dissolved and/or entrained carbon monoxide in the liquid reaction composition withdrawn from the first reaction zone is consumed in the second reaction zone
12. A process according to any one of claims 1 to 11 wherein methanol and/or methyl acetate are carbonylated with carbon monoxide in the first reaction zone.

13. A process according to any one of claims 1 to 12 wherein the concentration of methyl acetate in the liquid reaction compositions in the first and second reaction zones are independently in the range 1 to 70 % by weight.
14. A process according to any one of claims 1 to 13 wherein the concentration of
5 water in the liquid reaction compositions in the first and second reaction zones are independently in the range 0.1 to 20% by weight.
15. A process according to any one of claims 1 to 14 wherein the concentration of methyl iodide co-catalyst in the liquid reaction compositions in the first and second reaction zones is independently 1 to 20% by weight.
- 10 16. A process according to any one of claims 1 to 15 wherein the concentration of iridium carbonylation catalyst in the liquid reaction compositions in the first and second reaction zones is independently in the range 100 to 6000 ppm.
17. A process according to any one of claims 1 to 16 wherein the ruthenium promoter is present in the liquid reaction compositions in the first and second reaction
15 zones at a molar ratio of each ruthenium promoter : iridium in the range [0.1 to 100] : 1.
18. A process according to any one of claims 1 to 17 wherein the ruthenium promoter is present in the liquid reaction compositions in the first and second reaction zones at a molar ratio of each ruthenium promoter : iridium in the range [up to 10] : 1
19. A process according to any one of claims 1 to 18 wherein the concentration of
20 ruthenium in the liquid reaction compositions in the first and second reaction zones is independently less than 6000 ppm.
20. A process according to any one of claims 1 to 19 wherein the concentration of ruthenium in the liquid reaction compositions in the first and second reaction zones is independently 400 to 5000 ppm.
- 25 21. A process according to any one of claims 1 to 20 wherein the partial pressure of carbon monoxide in the first and second reaction zones is independently in the range 1 to 70 bar.
22. A process according to any one of claims 1 to 21 wherein the partial pressure of carbon monoxide in the first and second reaction zones is independently in the range 1
30 to 15 bar.
23. A process according to any one of claims 1 to 22 wherein the concentration of carbon monoxide in the low pressure off-gas is at least 30 mol% and the concentration of ruthenium in the liquid reaction composition is up to 3000 ppm by weight.

24. A process according to claim 23 wherein the concentration of carbon monoxide in the low pressure off-gas is 30 to 40 mol% and the concentration of ruthenium in the liquid reaction composition is up to 3000 ppm by weight.

25. A process according to any one of claims 1 to 22 wherein the concentration of carbon monoxide in the low pressure off-gas is at least 50 mol% and the concentration of ruthenium in the liquid reaction composition is up to 6000 ppm by weight.

26. A process according to claim 25 wherein the concentration of carbon monoxide in the low pressure off-gas is 50 to 60 mol% and the concentration of ruthenium in the liquid reaction composition is up to 6000 ppm by weight.

27. A process according to any one of claims 1 to 22 wherein the concentration of carbon monoxide in the low pressure off gas is at least 55 mol % and the ruthenium concentration in the liquid reaction composition is up to 5500 ppm by weight.

28. A process according to claim 27 wherein the concentration of carbon monoxide in the low pressure off gas is in the range 55 to 65 mol. % and the ruthenium concentration in the liquid reaction composition is up to 5500 ppm by weight.

29. A process according to any one of claims 1 to 28 wherein the process is operated as a continuous process.

30. Use of carbon monoxide in a low pressure off-gas, the carbon monoxide being maintained at a concentration in the low pressure off-gas according to the formula:

$$Y > mX + C$$

wherein Y is the molar concentration of carbon monoxide in the low pressure off-gas, X is the concentration in ppm by weight of ruthenium in the liquid reaction composition, m is about 0.012 and C is about -8.7, in a process for the manufacture of acetic acid which process comprises the steps of (1) carbonylating methanol and/or a reactive derivative thereof in a first carbonylation reaction zone in a liquid reaction composition comprising iridium carbonylation catalyst, ruthenium promoter, methyl iodide co-catalyst, methyl acetate, acetic acid and water;

(2) withdrawing liquid reaction composition together with dissolved and/or entrained carbon monoxide and other gases from said carbonylation reaction zone;

(3) optionally passing said withdrawn liquid reaction composition through one or more further reaction zones to consume at least a portion of the dissolved and/or entrained carbon monoxide;

- (4) passing said composition from step (2) and optional step (3) into one or more flash separation stages to form (i) a vapour fraction comprising condensable components and low pressure off-gas, the condensable components comprising acetic acid product and the low pressure off-gas comprising carbon monoxide and other gases dissolved and/or entrained with the withdrawn liquid carbonylation reaction composition and (ii) a liquid fraction comprising iridium carbonylation catalyst, ruthenium promoter and acetic acid solvent;
- (5) separating the condensable components from the low pressure off-gas; and
- (6) recycling the liquid fraction from the flash separation stage to the first carbonylation reaction zone,
- to reduce loss of catalyst and/or promoter during acetic acid product recovery.